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Effects of Cooling Rates on Microstructure, Wettability and
Strength of Sn3.8Ag0.7Cu Solder AlloyNadhras Murad^a, Siti Rabiattull Aisha^{b,*}, Mahadzir Ishak@Muhammad^{a,b,c},

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Abstract

The aim of this research paper is to study the effects of cooling rates on microstructure and mechanical properties of Sn3.8Ag0.7Cu solder alloy prepared through powder metallurgy (PM) method. They were cooled by different cooling medium such as slow (furnace cooling), normal (air cooling) and fast (water cooling) which was 0.0076°C/s, 0.73°C/s and 31.14°C/s, respectively. Characterisation for each sample was conducted to examine the intermetallic compound formation and solder shear strength. Result indicated that faster cooling rates decreased the IMC thickness but reduced the solder joint strength due to distribution of melted solder on Cu board. This study found that the shape of Cu₆Sn₅ and Ag₃Sn changed according to cooling rates either at the interfacial or in the solder matrix. The study on comparison between three different cooling rates onto properties of Sn3.8Ag0.7Cu solder alloy prepared by PM method is still unknown and the current findings are still unclear. Thus this research would be the fundamental study to investigate the effects of cooling rates onto behaviours of the solder alloy. It is believed that more attentions will be shown onto this superior topic to increase the level of promising reliability of solder alloy in industry and marketplaces.

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Keywords: cooling rate; Powder Metallurgy; solder; microstructure; intermetallic compound(IMC).

1.Introduction

Driven primarily by penetration of electronics usage into virtually every inches of life, everything on earth rely heavily upon electronics throughout the years. The diversity of application and never ending demand for both lower cost and higher performance cannot be achieved without major changes in materials and manufacturing processes.

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But improper metal like lead(Pb) in electronic parts has created public health concerns[1]. This issue has insists the industry to ensure the equally environmental sound of their products.

As for that, in the move to make product designs more environmental friendly, the regulatory trends such as Restriction of Hazardous Substances(RoHS), Joint Industry Guide(JIG), and some voluntary initiatives from nongovernmental organizations(NGOs) like Electronic Product Environmental Assessment Tool(EPEAT) officially affected products placed on the market world widely[2-5]. The use of Pb has been banned and therefore in the past few years, research and development involving governments, electronic producers and universities have spurred the study of lead free solder[4].

In term of manufacturing for lower cost of electronic products, a new technology in solder fabrication using Powder Metallurgy(PM) method is now ready to be scrutinized genuinely. All the equipment and machine involving PM method can be operated by minimum number, minimal waste and moderate skill of workers due to the manual operations involve[6, 7]. This for sure can benefit the company account profile. To begin, the solder pre mixed powder has to go through the milling, compacting and sintering as the basic process[3, 8, 9]. By virtue of the steps do not contain high temperature for fabricating the solder alloy, PM method also well known as green technology[10].

The solder alloy from PM method is likely to have better performance yet ensuring solder reliability. Literatures reported high functionality and affirmative results of solder fabricated by PM method but not as much are known about effects of cooling rate on those alloys and these have brought attention to conduct further research embracing cooling rate. Cooling rate plays an important understanding in solder alloy study as it has significant effects on the microstructure including solder joint interphase, solder matrix and IMC of solder as well as mechanical properties of the solder due to sensitivity of each element on processing and geometrical parameters[11, 12].

Common microstructures in SnAgCu solder alloy consist of primary β -Sn grains, platelet-type of Ag_3Sn and scallop-type of Cu_6Sn_5 [13]. In actual fact, the morphology of microstructures relies on cooling rate because here is the stage where the IMC get solidifies into distinctive shape, size and position then the strength of solder alloy can be predetermined[14, 15]. Yang has experimented Sn3.0Ag0.5Cu solder paste and using oven as reflow method. She reported that as the rate of cooling reduced, β -Sn dendrites coarsened, Cu_6Sn_5 had a thick stick-like, Ag_3Sn changed from branch-like to large platelet-like at the solder matrix and the IMC thickness also gets increased[16]. As the rate of cooling increased, Mueller found that needle-like Ag_3Sn became smaller in size, decreased spacing of β -Sn dendrites but no report on Cu_6Sn_5 and IMC thickness[15].

As a consequence to previous works above, the study different cooling rates on solder alloy is crucial to have better explanation in analysis to obtain specific properties and performance requirements of solder alloy. The Sn3.8Ag0.7Cu solder alloy used in this work performed as a fundamental study for the rest of solder alloy family prepared by PM method. Systematic evaluations on samples are conducted after reflow process to achieve melted solder to form joining on Cu board. The X-Ray Diffraction(XRD) assessment on the solder powder was a analysis to have information about phase formation of the solder after milling process.

2. Materials and methods

The experiment begun with some calculations and pre-weighed the Sn, Ag and Cu powder according to Sn3.8Ag0.7Cu with a analytical balance. It was then milled for 6 hours in a high energy planetary ball mill machine(Nian Hai Tianyang, FM-2 model) with a fixed speed of 1400rpm and 0.7g of it being compacted with 5 tons using a manual hydraulic press machine(Specac UK 15tons model) to make a solder pellet sample. There was no sintering process involved which made the samples as green bodies.

Before going through sample preparations process, samples were divided according to cooling effects such as slow, normal and fast cooling to assess the solder properties specifically after reflow process where the cooling effects would be occurred. 3 samples were reflowed on Cu board in a table top furnace(Nabertherm model N/11) with no protective gases up to 250°C for 25 minutes.

The first sample was let to cool in the furnace until it reach room temperature for slow cooling effect(0.0076°C/s), another one was taken out right after the reflowed processing time is completed for normal cooling effect(0.73°C/s) and the other one was cooled in a beaker filled with tap water until it reached room temperature for fast cooling effect(31.14°C/s). All those samples were then went through several analyses including wettability angle measurement, scanning electron microscope(SEM) and energy dispersive x-ray(EDX) for IMC characterisation and single-lap shear test for evaluating solder joint strength. The phase formation of milled Sn3.8Ag0.7Cu solder alloy was examine with X-Ray diffraction(XRD) analysis.

3. Results and discussion

3.1 XRD Analysis

Figure 1 depicted the XRD result and analysis for the milled Sn3.8Ag0.7Cu solder powder. The interference patterns interpreted some sharp, narrow and high intensity peaks which defined the phase's formation of intermetallic compound formed in the solder powder.

They were Ag_3Sn , Cu_6Sn_5 and $\beta\text{-Sn}$ since Sn is the major portion from the whole composition where it reacted with Cu substrate to yield such phases. This XRD result was compatible with the EDX analysis which reported below, where the same IMC were successfully detected.

With this XRD results, it could be concluded that the milled Sn3.8Ag0.7Cu solder powder was a crystalline structure.

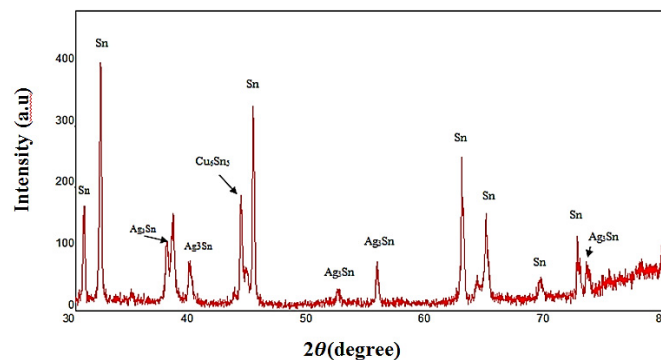


Figure 1: The XRD analyses on milled Sn3.8Ag0.7Cu solder powder

3.2 Wettability

Wettability is one of the joining measures for two mediums joined together where any liquid with a solid surface called substrate to stay intermolecular contact with each other. The liquid then have an ability to spread over the substrate and creating contact angle which represents wetting area and wetting angle, respectively.

Wetting angle could be studied by analysing the top surface and cross section of microstructure with the help of Scanning Electron Microscope(SEM).From the literatures, the wetting angle can be illustrated as in Figure 2.

The most favourable wettability of a solder is when the solder has large area of its molten spreading on substrate and having the lower of wetting angle. This is because, both of factors could lead to a high strength of adhesion to the substrate and prolonged the reliability of solder.

In terms of wetting angle, all the cooling rates that have been used on the compacted solder depicted a very good range of wetting angle as shown in Figure 3 where slow, normal and fast cooling rate had produced 14.43°, 10.92° and 11.96°, respectively. These values are compatible to other solder fabrication such as casting method. Based on these values, all cooling medium used were compatible to be used since they managed to produced good wetting on the copper board.

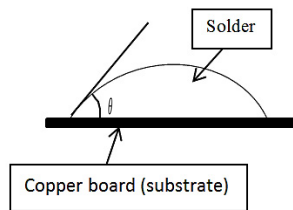


Figure 2: Illustration of wetting angle

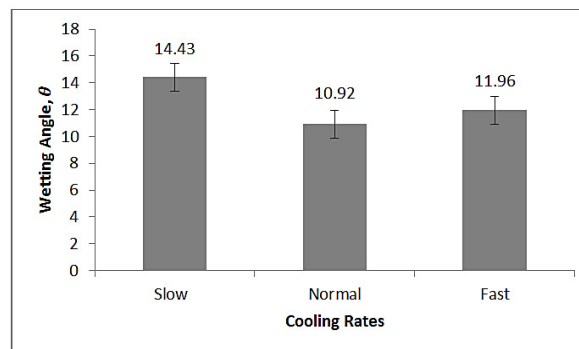


Figure 3: Data of wetting angle according to different cooling rates

3.3 Intermetallic Compound(IMC)Formation

Results indicated that the temperature of 250°C used for 25 minutes was suitable for this work where it managed to melt the solder pellet by triggering the IMC to form and allowed different cooling rates to take reactions.

Figure 4a, 4b and 4c expounded the images of IMC cross section at the interfacial of Cu substrate according to their respective cooling rates. It has been reported by many researches that SnAgCu solder based will mainly comprised with Cu_6Sn_5 phases where the Sn atom has responded to Cu substrate to form those IMC phases.

The normal cooling showed the scallop type of Cu_6Sn_5 IMC at the interfacial as in Figure 4b. This type of IMC morphology was the result of Cu atoms from the substrate diffused into molten solder. Due to slow cooling in the furnace, the morphology of Cu_6Sn_5 at the interphase was scallop type at first before turned into planar type as in Figure 4a and because of the aging process and factor of grain-regrowth, the Ag_3Sn had enough time to become a large plate-like shape from particle-like in the Sn matrix.

The fast cooling rate yielded non-equilibrium phase where no presence of Cu_6Sn_5 or Ag_3Sn could be detected in the solder matrix because of low diffusion rates of Ag and Sn atoms on the solder matrix as can be seen in Figure 4c but noted the Cu_6Sn_5 phases were still at the interfacial due to reflow process. The matrix is filled with β -Sn dendrites instead.

This depicted, Cu_6Sn_5 at the interfacial existed during reflow while Cu_6Sn_5 existed during solidification. Therefore, through these microstructure analyses, it is understandable that the cooling rate does affecting the evolution of solder microstructure during and after the reflow process.

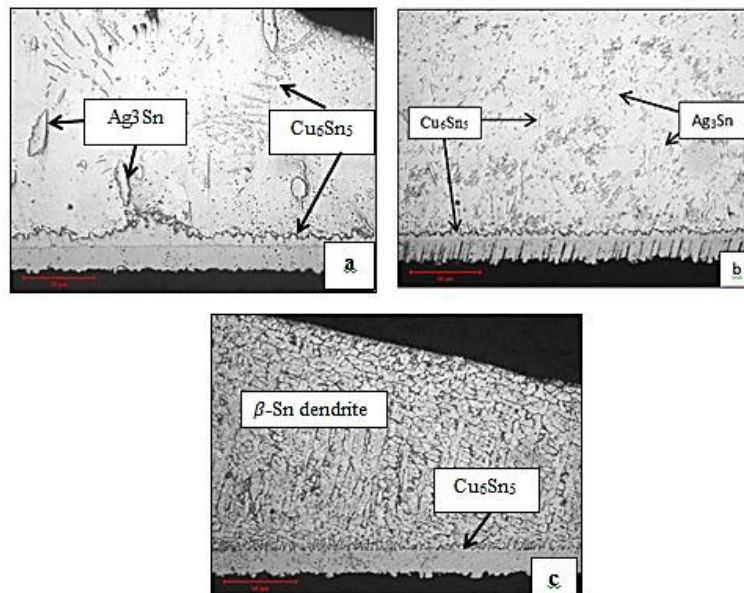


Figure 4: Cross sections of IMC by respect to (a) slow, (b) normal, (c) fast cooling

3.4 Thickness of intermetallic compound (IMC)

The study of IMC thickness is so much crucial because it indicates the solder characterisation by knowing the value. Theoretically, the higher the IMC thickness, the weaken the solder strength because IMC properties itself is brittle in nature[17]. Using the Image-J Software to measure the thickness of interfacial IMC, five cross sectional images were randomly selected from those three cooling rates and the averages of thicknesses were graphed like in Figure 5.

It can be seen clearly that slow cooling rate had the highest IMC thickness which due to short aging treatment and this had allow the Cu_6Sn_5 phase to grow up in timely manner. But no Cu_3Sn was detected as the slow cooling temperature kept decreasing by time until the furnace reached room temperature so it did not enough time and temperature to be existed in the layer.

It is a closed comparison for normal and fast cooling rates where the different is just about $0.48\mu\text{m}$ which showed that both of cooling rates could be the best choice to use for solder pellet. But care to note that fast cooling rates yielded large distribution of coarsed β -Sn dendrite on solder matrix which lead to low strength of solder because of the brittle property.

Thus, by analyzing the IMC thickness result, it is fair to choose normal cooling as the favorable cooling method to cool down the solder pellet in order to maintain solder reliability.

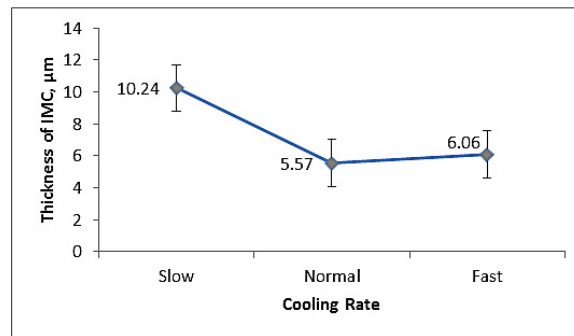


Figure 5: Thickness of intermetallic compounds by according to slow, normal and fast cooling

3.5 Single Lap-shear strength

Highest shear strength was dominated by slow cooling sample followed by normal and fast cooling as illustrated in Figure 6. Knowing that shear strength from slow cooling had the highest value of IMC thickness, this is somehow a fascinating situation to explore the reason behind.

Owing to the fact that higher IMC leads to the weak strength[6], clearly that factor did not work with this type of solder alloy prepared from PM method where slow cooling revealed the highest value of shear strength which is 29.65MPa . But there is one factor to be considered when it comes into PM compact solder which is the diameter of pellet used in this work distinctly.

Figure 7 depicted this complication where 13mm of solder diameter cannot be fully melted on the Cu board as molten solder should be. It is understandable that large distribution of melted solder on Cu board rendered higher shear strength. This could be the reason of higher shear strength value for slow cooling sample.

With normal cooling, the shear strength recorded a value of 29.01MPa whereas fast cooling depicted the lowest value of shear strength in this experimental work which was 12.66 MPa. This is to be confirmed that the shear strength result of fast cooling sample was a factor from IMC thickness and spreadable melted solder on Cu board.

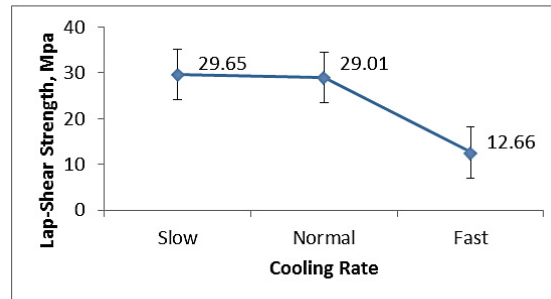


Figure 6: The value of shear strength according to cooling rates



Figure 7: The actual condition of compacted Sn3.8Ag0.7Cu solder alloy after lap shear strength test

CONCLUSIONS

It is obvious to see that different cooling rates give different results to all analyses on the samples. These proven that the study of cooling rate is so much crucial in order to have a specific yet high reliability of solder. Thus the experiment results supported following conclusions:

- The XRD result depicted a crystalline structure of milled solder alloy before continue with pressing method. This has verified that the PM method successfully assemble each element to form homogeneous mixture of solder alloy.
- The scallop-type of Cu_6Sn_5 still showed up at the interfacial solder joint for all cooling rates but as the cooling rate reduced, it changed into planar-type of shape due to aging effect. The IMC in the solder matrix depicted different shape of microstructure of Ag_3Sn for slow cooling and large β -Sn dendrites detected all over the cross sections of solder matrix for fast cooling.
- As the cooling rate reduced, thickness of IMC seemed to be higher. This was due to aging effect upon slow cooling rate occurred in furnace.
- Another resolution shall be made in this work in order to have a better distribution of molten solder alloy to be fully spreadable on Cu board to achieve maximum shear strength.
- Thus, Powder Metallurgy(PM) method could serve as a promising and new green technology to fabricate lead-free solder alloy for the brighten future of electronic industry.

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